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(54) Stabilization of liquid coffee by treatment with alkali

(57) The present invention is directed to a liquid coffee in which the development of acidity has been inhibited and which results in a longer shelf-stable product. The method used in making the liquid coffee product of the present invention comprises treating the coffee extract with an alkali, said alkali being present in an amount effective to convert acid precursors present in the coffee extract to their respective acid salts, and thereafter neutralizing the treated coffee extract with an acid, said acid being present in an amount effective to neutralize any excess alkali from the first step and to adjust the final pH of the liquid coffee product to the desired value.

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Description

BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION

The present invention is generally related to the field of coffee, and more particularly to the making of an improved liquid coffee. More specifically, the present invention is directed to a technique of stabilizing a liquid coffee product by inhibiting the development of acidity which occurs on storage. This is accomplished by treating the coffee extract with an alkali and thereafter neutralizing the solution to the desired pH. The alkali treatment hydrolyzes the lactones and esters which are present in solubilized coffee solids and converts them into stable salts. The lactones and esters are the acid precursors which are responsible for the increase in acidity of stored liquid coffee products.

DESCRIPTION OF THE PRIOR ART

The acidic nature of coffee and the role it plays in the ultimate quality of a coffee beverage is well documented. Although the acid fraction of a coffee extract generally constitutes from only about 10 to about 15% of the solids in the extract, the effect that acids have on the final coffee product can be said to be a determining factor in the consumer appeal of a coffee beverage. More particularly, too much acidity in a coffee can result in an undesirable sourness to the beverage, while too little acidity in the coffee causes a "flat" flavor profile. Finding and maintaining the right acidic balance is critical.

The task of adjusting the acidity of a coffee beverage is not made any easier by the fact that well over 25 different acids have been identified in roasted coffee. More particularly, about 8% of green coffee is comprised of numerous isomers of caffeoylquinic acids, also referred to as chlorogenic acids. Other principal acids found in green coffee include malic and citric acids. Roasted coffee produces additional acids, such as acetic, formic, glycolic, lactic and pyroglutamic acids.

Various factors are known which affect the acidity of a coffee extract. For example, different bean varieties result in varying acidity. The pH of a coffee brewed from Arabica varieties is generally found to be between 4.85 and 5.15. In contrast, coffee brewed with Robusta beans generally have a pH in the range of 5.25 to 5.40. Other factors which are said to influence the degree of acidity include the degree of roast, the type of roaster, the nature of the processing and the age of the green beans.

Liquid coffee products, although not widespread in the United States, represent a significant part of the Japanese and Korean coffee markets. The product is usually pre-sweetened and ready to drink. The liquid coffee product is generally prepared by mixing a diluted coffee extract with the desired additives, such as milk, sugar and flavorants. The product is then packaged in a suitable container, such as a can, which can be subjected to retort processing. The result is a liquid coffee product which can be distributed to the consumer. The product can be stored for generally up to six months at room temperature before consumption.

Unfortunately, a major problem exists with the marketing of liquid coffee. More particularly, coffee extract is an unstable system and both the shelf- and refrigerator-stored liquid coffee products currently available develop an increased acidity over a short period of time. Too low of a pH also results in the possible curdling of the milk or cream. Simply, the rise in acidity translates into a loss of quality to the product. That is, there is an increased sourness to the liquid coffee product. This quality loss is known as "staling" and although the cause of staling is attributed for the most part to the drop in pH and the increase in titratable acidity, no clear explanation or mechanism is known for its occurrence.

H.G. Maier, et al., Dtsch. Lebensmittel-Rdsch. 80(9): 265-268 (1984) have shown that the content of low molecular weight acids increase on storage at elevated temperatures and attributed the increase to the hydrolysis of esters and lactones produced on roasting.

One solution which has been used to prevent the problem of sourness development is the addition of sodium bicarbonate to elevate the initial pH of the product. However, the product pH of the sodium bicarbonate-treated liquid coffee product still falls on storage and has additional potential repercussions on product flavor.

Consequently, there is still a need for a process that prevents the accumulation of acidity in stored coffee extracts.

SUMMARY OF THE INVENTION

The process of the present invention was developed to find a solution to the staling of liquid coffee extracts. The fact that there is a concomitant increase in titratable acidity in stored coffee extracts is indicative of acid being generated during storage. This in turn points to the presence of a significant quantity of acid precursors in fresh coffee extracts. By the use of the treatment described herein, liquid coffee can be stored without deterioration of quality.

Specifically, the present invention is directed to a method for stabilizing a coffee extract comprising the steps of treating the coffee extract with an alkali, said alkali being present in an amount effective to convert acid precursors present in the coffee extract to their respective acid salts, and neutralizing the treated coffee extract with an acid, said acid being present in an amount effective to neutralize any excess alkali and obtain a final pH of the coffee extract of from about 4.8 to about 5.2.

The stabilization treatment of the present invention offers the opportunity to extend the product shelf-life of liquid coffee products and consequently make them more appealing to the consumer.

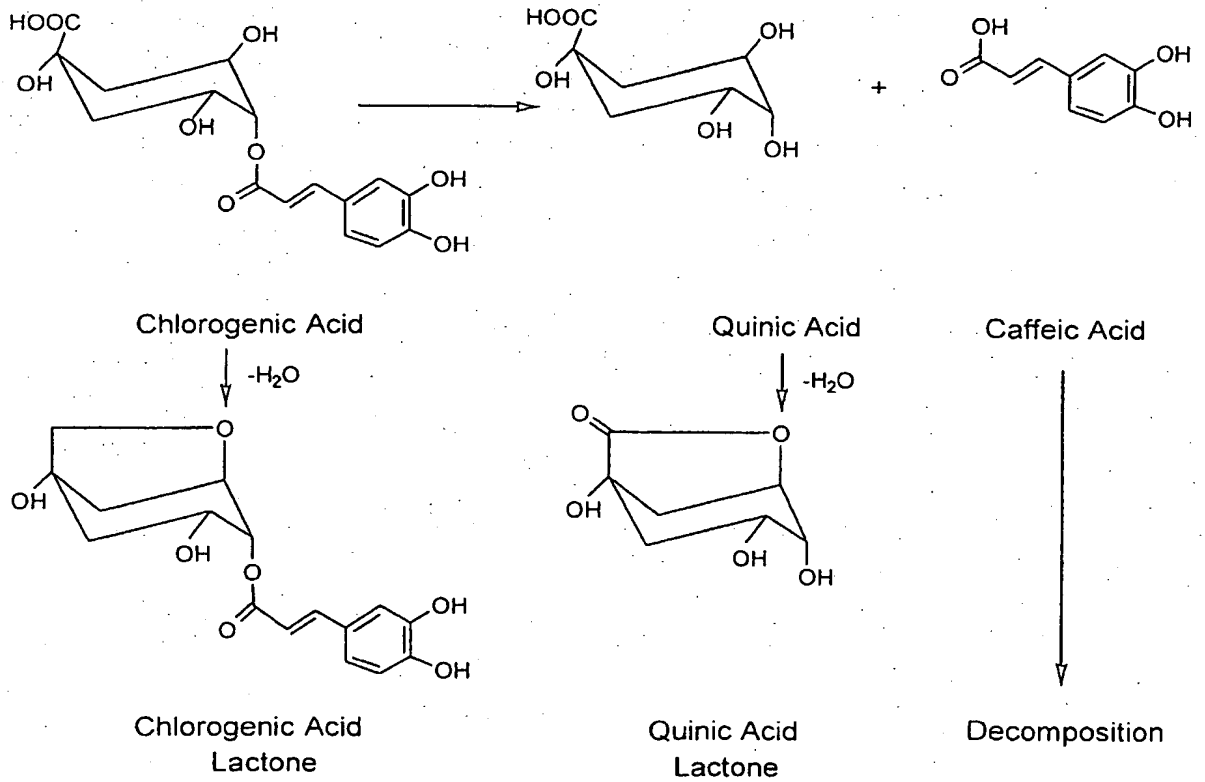
The present invention is further directed to an improved liquid coffee product made in accordance with the stabilizing treatment described above. The liquid coffee product made in accordance with the present invention possesses a longer and more stable shelf-life than any known liquid coffee product currently available.

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a method for inhibiting the development of acidity which occurs during storage of a coffee extract. In order to suppress the acidity increase in stored coffee, it was first necessary to study the changes in the concentrations of organic acids during storage of a coffee brew and to identify the acids which contributed most to the increase in acidity.

As is evidenced by the data obtained in Comparative Example 1, it was found that the major contribution to increased acidity in a stored coffee brew was provided by the production of quinic acid, which increased by almost 40%. Other acids which showed significant increases in concentration were acetic acid (24%), glycolic acid (16%), formic acid (14%) and phosphoric acid (27%). Citric and malic acids showed no significant increase, while the other acids being monitored showed small increases.

Once the major acids were identified which were responsible for the acidity increase in the stored coffee, the next step in the development of the present invention was to understand the reactions which led to the production of these acids. For example, upon roasting, it has been found that both quinic and chlorogenic acids form lactones, i.e. quinic acid lactone and chlorogenic acid lactone. Their formation can be illustrated as follows:



As shown above, the chlorogenic acid present in the coffee degrades on roasting to produce both quinic acid and chlorogenic acid lactone. As is further illustrated, the quinic acid then breaks down to produce quinic acid lactone. The present invention realizes that these lactones represent the primary precursors to the acids which develop upon storage. The gradual hydrolysis of the above-noted lactones are responsible for the development of chlorogenic acid and quinic acid in untreated liquid coffee.

Through the use of gas chromatography and mass spectroscopy, it was determined that in a stored coffee brew (for a period of 8 days at a temperature of 60°C), the concentration of quinic acid increased by 14.8 mmol/kg while the quinic acid lactone concentration decreased by 12.2 mmol/kg. This translates into a 29.6% increase in acid and a 24.5% decrease in the lactone. The large increase in quinic acid concentration on storage suggests that the hydrolysis of the quinic acid lactone is an important contributor to the acidity development.

Similarly, it was determined that based on the existence of the chlorogenic acid lactones in the stored coffee, the hydrolysis of the lactones to chlorogenic acid represented a 10 to 15% increase in the overall acidity of the coffee on storage.

The other acids which were found to increase over time, such as acetic acid, formic acid, phosphoric acid and glycolic acid, are all low molecular weight acids which are believed to be produced on storage by hydrolysis of precursor esters. It is suggested that these precursor esters could be produced by reaction of acids produced during roasting with hydroxyl groups such as those present in the coffee polysaccharides. On hydrolysis, the acids would then be released. Increases of about 20%, 10%, 7% and 6% were found to occur with acetic acid, formic acid, phosphoric acid and glycolic acid, respectively. Overall, hydrolysis of esters produced on roasting are believed to be ultimately responsible for about 35% of the acidity increase in the stored liquid coffee product. These are percentages of the total acid increase.

In addition to the above, it is further noted that polymeric Maillard-type products known as coffee melanoidins are believed to contribute to the sourness of roast coffee. Based on the fact that coffee melanoidins are acidic and contain a variety of functional groups, it seems likely that they would also contain ester or lactone linkages which would contribute to acidity increase on storage.

Based on the observations described above, the overall acidity increase in a stored coffee extract was found to be due primarily to the formation of acids by hydrolysis of esters and lactones produced on roasting. Based on these findings, a method was developed for inhibiting acid production and thus stabilizing a coffee extract such that staling of the coffee product would not occur.

In the process of the present invention, the first step in stabilizing a coffee extract is the treatment of the coffee extract with an alkali. Alkalies are compounds that contain the hydroxide ion (OH). When the alkali reacts with an ester a saponification reaction occurs which converts the ester group into an acid salt and an alcohol group. Thus, in order to prevent the lactones and esters from forming acids by hydrolysis during storage and increasing the acidity of the beverage, the alkali is reacted with the lactones and esters to produce the stable acid salts. Accordingly, on storage, the lactones and esters are no longer present and cannot form their acid counterparts through hydrolysis.

The amount of alkali to be added to the extract in the process of the present invention must be in a sufficient quantity to convert the acid precursors present in the coffee extract into their respective acid salts. Based on the fact that different varieties of coffee and degrees of coffee roast are comprised of different percentages of acids, the required amount of alkali will vary depending on the coffee blend which is used, as well as other factors which effect the acid composition of the coffee extract. Generally speaking, however, the alkali is added in an amount from about 0.1 mol/l to about 0.5 mol/l. A preferred amount of alkali is from about 0.25 mol/l to about 0.35 mol/l.

Alkalis as defined herein may be any of those typically used in the art and include any food-acceptable alkalis such as sodium hydroxide, calcium hydroxide, potassium hydroxide and the like. Potassium hydroxide is the preferred alkali because potassium is naturally present in coffee and it is less detectable from a flavor perspective.

The treatment of the coffee extract in the first step of the method of the present invention should be conducted at a temperature and pH suitable for the neutralization reaction to take place. The time for the neutralization reaction, i.e. the conversion of the lactones and esters into their respective stable acid salts, will also vary depending on the other variables. More specifically, use of a lower pH is possible at elevated temperatures. For example, the pH of the coffee extract may be raised to a pH of 10 by the addition of N KOH at room temperature for one hour. Alternatively, the pH can be held at 9 if the coffee extract is treated at a temperature of 60°C for the same time period. Generally, the coffee extract can be treated in the temperature range of 0°C to 80°C. The higher temperatures allow the use of low pH (as low as 8.5) and short processing times (as short as 1 minute). Lower temperatures would require high pH (as high as 12.0) and longer processing times (as long as 1 day). The adjustment of these variables to ensure the reaction with the alkali is well within the abilities of those skilled in the art.

Because of primarily economic considerations, there are two preferred set of processing conditions to effect alkali treatment of the coffee extract. The first preferred set of conditions will treat the coffee extract at room temperature at from about 20°C to about 25°C. At these temperatures, the extract should be maintained at a pH of from about 9.5 to about 12 for a time effective to convert acid precursors to their respective acid salts. The preferred time should vary

from about 0.75 to about 1.25 hours. The second set of conditions will treat the coffee extract at elevated temperatures of from about 55°C to about 65°C. At these temperatures, the extract should be maintained at a pH of from about 8.8 to about 9.5 for a time effective to convert acid precursors to their respective acid salts. The preferred time should vary from about 0.75 to about 1.25 hours.

The second step in the method of the present invention is the neutralization of the treated coffee extract resulting from the first step. After the first step has been completed, excess alkali is present and the pH of the extract is too high. By addition of an acid, the excess alkali is neutralized and the pH can be adjusted to the desired value.

Acids which may be used in the present invention may be any of those typically used in the art and include any food-acceptable acid such as various types of phosphoric acid, citric acid, tartaric acid, fumaric acid, adipic acid, malic acid and the like.

Of course, the specific amount of acid to be used, and the type of acid to be used, will depend on the desired qualities of the end product, specifically the desired pH as well as desired flavor. That is, an acid is added to obtain a final pH which results in an optimum sensory quality. Typically, for liquid coffee beverages, a pH of from about 4.7 to about 5.3 is desired, with a pH of about 4.9 to about 5.1 being preferred.

Alternatively, the neutralization reaction of the second step of the present invention may be performed with the use of a cation exchanger in the [H^+] form. The specifics of such reactions are well known to those skilled in the art and do not need to be detailed.

The coffee extracts treated by the method of the present invention were found to have a low quinic acid lactone content indicating what the results would eventually show with respect to the storage of the coffee over time. As was expected, the liquid coffee extracts showed no appreciable drop in pH. A few minor changes were detected in the composition of the coffee volatile fraction, however, the product after a storage time equivalent to six months at room temperature had only a slightly reduced flavor intensity as compared to a fresh control sample. Moreover, the preferred process seeks to avoid loss of aromatics by removing aromatics from the extract prior to alkali treatment, as for example by steam distillation and then adding back these aromas after neutralization of the alkali-treated extract. The increased salt level in the treated extract was detected by a few, but not all, tasters on the taste panel.

The liquid coffee product produced by the method of the present invention exhibits a longer and more stable shelf-life than liquid coffee products currently available in the market. Due to the stabilized pH, there is no longer a risk of milk flocculation occurring on storage. As indicated above, the liquid coffee products made in accordance with the present invention are characterized by either the absence of or having a very low level of quinic acid lactone. Generally, liquid coffee products treated by the method of the present invention will have less than 0.05% quinic acid lactone content.

The following examples are provided to further illustrate the present invention.

COMPARATIVE EXAMPLE 1

Based on reaction kinetics which show a strong temperature dependence, it was determined that for acid formation, storage of a coffee extract at a temperature of 25°C for a period of six months was equivalent to the storage of the same extract for a period of 14 days at a temperature of 60°C.

A standard coffee solution made from Colombian beans at a temperature of 60°C was stored and monitored over a 14 day period. After about 200 hours, the development of acid had leveled off, with the pH dropping from about 4.9 to about 4.5. The pH drop resulted in an unpleasant sour taste. The resulting data (see Table 1 below) showed an increase of several organic acids.

Table 1:

Change in organic acids in stored coffee brew						
Acid	Time [hrs]					
[g/kg]	0	2.5	8	24	72	120
Quinic	7.8	8.7	8.7	9.0	9.9	10.8
Acetic	3.15	3.6	3.6	3.6	3.9	3.9
Glycolic	1.14	1.29	1.23	1.25	1.23	1.32
Formic	2.0	2.10	2.13	2.19	2.22	2.28
Malic	2.09	2.19	2.16	2.40	2.22	2.19
Citric	6.6	6.9	6.9	6.9	6.9	6.9
Phosphoric	1.44	1.50	1.53	1.59	1.71	1.83

Example 1

R & G coffee was extracted with hot water to give an 8% liquid coffee solution. The extract was treated with N KOH with stirring so that the pH was maintained at a value of 10 at room temperature for a time of one hour. The solution was then neutralized to a pH of 4.8 using 85% H_3PO_4. Storage studies (for a period of 14 days at a temperature of 60°C) showed no drop in pH. The product after the storage period was comparable in taste to the product described in Example 1.

Although some minor changes occurred in the composition of the coffee volatile fraction, optimization of conditions for the treatment of the coffee extract resulted in a product which had only slightly reduced flavor intensity when compared to a fresh control sample.

Example 2

R & G coffee was extracted with hot water to give an 8% liquid coffee solution. The extract was treated with 10N KOH with stirring, and at a temperature of 60°C, so that a pH was held at a value of about 9.0. The treatment was for approximately one hour.

The solution was then neutralized to a pH of 5.0 using 85% H_3PO_4. The solution was stored for a period of 14 days at a temperature of 60°C. At the end of the storage period, the product showed no observable drop in pH and had a comparative flavor to a fresh control sample.

Example 3

R & G coffee was extracted with hot water to give an 8% liquid coffee solution. The extract was treated with 10N NaOH with stirring so that the pH was held at above pH 12 at room temperature for 1 hour. The solution was then neutralized to a pH of 4.73 using 85% H_3PO_4. Following the neutralization step, storage studies showed that the observed drop in pH as was shown in Comparative Example 1 no longer occurred. The solution pH remained stable on storage (60°C, 8 days).

Alkali extract treatment at pH 12 requires higher phosphoric acid addition to effect neutralization as compared to the previous two examples. As a result, this will increase the likelihood of sensory perception of the generated phosphate salt in the treated extract.

The above preferred embodiments and examples are given to illustrate the scope and spirit of the present invention. The embodiments and examples described herein will make apparent, to those skilled in the art, other embodiments and examples. These other embodiments and examples are within the contemplation of the present invention. Therefore, the present invention should be limited only by the appended claims.

Claims

1. A method for stabilizing a coffee extract comprising the steps of:

   a) treating the coffee extract with an alkali, said alkali being present in an amount effective to convert acid precursors present in the coffee extract to their respective acid salts; and
   b) neutralizing the treated coffee extract of step (a) with an acid, said acid being present in an amount effective to neutralize any excess alkali and obtain a final pH of the coffee extract of from about 4.7 to about 5.3.

2. A method according to claim 1, wherein said alkali is a food-acceptable alkali.

3. A method according to claim 2, wherein said food-acceptable alkali is potassium hydroxide.

4. A method according to any one of claims 1 to 3, wherein the effective amount of said alkali ranges from about 0.1 mol/l to about 0.5 mol/l.

5. A method according to claim 4, wherein the effective amount of said alkali ranges from about 0.25 mol/l to about 0.35 mol/l.

6. A method according to any one of claims 1 to 5, wherein said acid precursors are lactones and esters.

7. A method according to claim 6, wherein said lactones are chlorogenic acid lactone and quinic acid lactone.

8. A method according to any one of claims 1 to 7, wherein step (a) is conducted at a temperature of from about 20°C to about 25°C.

9. A method according to claim 8, wherein said extract is maintained at a pH of from about 9.5 to about 12.0 for a time effective to convert said acid precursors to their respective acid salts.

10. A method according to claim 9, wherein said effective time is from about .75 to about 1.25 hours.

11. A method according to any one or claims 1 to 7, wherein step (a) is conducted at a temperature of from about 55°C to about 65°C.

12. A method according to claim 11, wherein said extract is maintained at a pH of from about 8.8 to about 9.5 for a time effective to convert said acid precursors to their respective acid salts.

13. A method according to claim 12, wherein said effective time is from about .75 to about 1.25 hours.

14. A method according to any one of claims 1 to 13, wherein said acid is a food-acceptable acid.

15. A method according to claim 14, wherein said acid is selected from phosphoric acid, citric acid, fumaric acid, malic acid, tartaric acid and adipic acid.

16. A method according to claim 15, wherein said food-acceptable acid is phosphoric acid.

17. A method according to any one of claims 1 to 15, wherein said final pH is from about 4.7 to about 5.3.

18. A method according to claim 17, wherein the pH ranges from about 4.9 to about 5.1.

19. A method according to any one of claims 1 to 13, wherein step (b) comprises neutralizing the treated coffee extract of step (a) with the use of a cation exchanger in the [H^+] form.

20. A method according to any one of claims 1 to 19, further comprising removing aromatics from the extract prior to step (a) and adding back said aromatics to the treated extract after step (b).

21. A liquid coffee composition made by a method according to any one of claims 1 to 20.

22. A liquid coffee composition according to claim 21, wherein said composition comprises less than 0.05% quinic acid lactone.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 98 30 0217

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	GB 439 017 A (H. MERKEL) * page 1, line 90 - line 97; claims 1-6 *	1-8,21, 22	A23F5/24 A23F5/18
A	FR 2 454 273 A (SOCIETE DES PRODUITS NESTLE) * page 1, paragraph 2 *	1-3	
A	CH 367 699 A (A. HAGEN) * claim 1; example *	1	
A	US 3 644 122 A (J. YERANSIAN) * column 1, line 30 - line 75 *	1-7	
A	DE 568 821 C (P. JACOB) * claim *	1	
A	US 3 753 726 A (W. CLINTON) * column 1, line 26 - line 33; claims 1W.,C1 *	1,15	
A,D	H. MAIER: "Säuren des Kaffees" DEUTSCHE LEBENSMITTEL-RUNDSCHAU, vol. 80, no. 9, 1984, pages 265-268, XP002062292 * page 267, column 2 *	1,2,6,7, 20,21	TECHNICAL FIELDS SEARCHED (Int.Cl.6) A23F
A	PATENT ABSTRACTS OF JAPAN vol. 096, no. 011, 29 November 1996 & JP 08 173043 A (YUNIKAFUE:KK), 9 July 1996, * abstract *	1	
A	J. HUCKE: "Chinasaürelacton im Kaffee" LEBENSMITTEL UNTERSUCHUNG UND FORSCHUNG, vol. 180, no. 5, 1985, pages 479-484, XP002062293		

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	17 April 1998	Desmedt, G

EPO FORM 1503 03.82 (P04C01)

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 98 30 0217

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	US 4 076 855 A (W. RYDER)		

TECHNICAL FIELDS SEARCHED (Int.Cl.6)

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	17 April 1998	Desmedt, G

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& : member of the same patent family, corresponding document

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(54) Beverage antioxidant system

(57) An antioxidant system for ready-to-drink beverages and beverage concentrates. The antioxidant system is particularly suitable for coffee beverages. The antioxidant system is made up of glucose oxidase, a glucose oxidase substrate, a catalase and an inorganic oxygen scavenger. The beverages have improved aroma and flavour.

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Description

[0001] This invention relates to an antioxidant system for beverages; especially beverages in ready-to-drink form. The invention also relates to beverages and beverage precursors which contain the antioxidant system and to processes for removing oxygen using the antioxidant system.

[0002] Many beverages suffer adverse effects from exposure to oxygen. This is particularly the case with ready-to-drink beverages; especially ready-to-drink coffee beverages. Ready-to-drink coffee beverages are produced by extracting soluble coffee solids from roasted and ground coffee beans using hot water. The extract obtained may then be diluted to a desired concentration, usually to contain about 1% by weight of soluble coffee solids. Various additives are added to the diluted extract which is then filled into containers. The containers are then sealed and subjected to retorting. Certain intermediate steps may also be carried out. For example, the extract may be concentrated and dried to powder prior to formation of the dilute extract. This is usually done when the coffee is filled into the containers at a site different than the site at which the extraction is carried out.

[0003] During this process, the coffee may be exposed to oxygen several times. For example, oxygen may be present in the hot water which is used to extract the soluble coffee solids from the roasted and ground coffee beans. Also, the coffee may be exposed to oxygen during extraction or subsequent processing such as concentration and drying. Further, oxygen may get into the container during filling. No matter where in the process the coffee is exposed to oxygen, it is now recognised that the oxygen adversely effects the flavour and aroma of the coffee beverage. In particular, the beverage loses its fresh, clean flavour and aroma; the flavour and aroma which characterises freshly brewed coffee. Often, bitter, acid flavours develop.

[0004] Various measures have been taken in the past to reduce the influence of oxygen. Usually these methods have centred on preventing ingress of oxygen. For example, Japanese patent application 6-141776 discloses extracting coffee grounds using deoxygenated water in an inert gas atmosphere. Further, all subsequent steps, including filling of the dilute extract into containers, is done under inert gas atmosphere. The patent application describes the resulting product to have a good, fresh flavour. The inert gas recommended is nitrogen. The primary problem with this technique is its cost. Carrying out an entire extraction and filling process in a nitrogen gas atmosphere is extremely expensive. Also, deoxygenating water is not a perfect process and not all oxygen is removed.

[0005] Another approach which has been attempted is to use antioxidants during the process. For example, US patent 5,384,143 describes a process in which the coffee extract is rapidly cooled to below 20°C and then an antioxidant selected from erythorbic acid, ascorbic acid, and their water soluble salts, is added to the cooled extract. The extract is then filled into cans under oxygen free conditions. This technique is less expensive than carrying out the entire process under inert gas atmosphere but there are problems. In particular, coffee is a potent antioxidant which is able to scavenge oxygen faster than most antioxidants commonly used in foods. Therefore, although the antioxidants described in this patent remove some of the oxygen, they are not potent enough to prevent the coffee from scavenging a large portion of the oxygen present. Consequently, the coffee undergoes some oxidative damage.

[0006] A further approach has been the use of enzyme systems. For example, the use of systems based upon glucose oxidase and alcohol oxidase have been suggested. However these systems have not proved to be adequate since degradation due to oxygen still occurs. Also, these enzyme systems often produce hydrogen peroxide which is undesirable.

[0007] Therefore it is an object of this invention to provide an antioxidant system which is relatively inexpensive and which is sufficiently potent to remove oxygen from beverage components which are themselves antioxidants.

[0008] Accordingly, in one aspect, this invention provides an antioxidant system for ready-to-drink beverages, the system comprising glucose oxidase, a catalase, a glucose oxidase substrate and an inorganic oxygen scavenger.

[0009] It has been surprisingly found that the combination glucose oxidase, a catalase, a glucose oxidase substrate and an inorganic oxygen scavenger is a sufficiently potent antioxidant such that small amounts are able to adequately compete with beverage components which are potent antioxidants, such as coffee. Since small amounts are required, the system therefore offers the advantage of being an inexpensive and effective antioxidant. Also, the system is food grade; especially at the small amounts required.

[0010] In another aspect, this invention provides a ready-to-drink beverage which includes an antioxidant system, the system comprising glucose oxidase, a catalase, a glucose oxidase substrate and an inorganic oxygen scavenger.

[0011] The ready-to-drink beverage is preferably a coffee beverage; especially a black coffee beverage. The ready-to-drink beverage may be retorted.

[0012] In a yet further aspect, this invention provides a beverage concentrate which includes an antioxidant system, the system comprising glucose oxidase, a catalase, a glucose oxidase substrate and an inorganic oxygen scavenger.

[0013] The inorganic oxygen scavenger is preferably a sulphite; for example sodium sulphite.

[0014] In another aspect, this invention provides a process for reducing oxygen in a beverage, the process comprising:

adding an antioxidant system comprising glucose oxidase, a catalase, a glucose oxidase substrate and an inorganic oxygen scavenger to the beverage;
filling the beverage into containers; and
sealing the containers.

[0015] In another aspect, this invention provides a process for reducing oxygen in a beverage containing extracted solids, the process comprising:

adding an antioxidant system comprising glucose oxidase, a catalase, a glucose oxidase substrate and an inorganic oxygen scavenger to an extraction liquid;
extracting solids from an extraction substrate using the extraction liquid to provide a beverage;
filling the beverage into containers; and
sealing the containers.

[0016] Preferably, the beverage is filled into containers under oxygen reduced or oxygen free conditions. Further, further amounts of the antioxidant system may be added to the beverage prior to sealing of the containers.

[0017] Embodiments of the invention are now described, by way of example only. This invention provides an antioxidant system which is useful for removing oxygen from beverages and beverage concentrates. The antioxidant system may be used, for example, during the processing of the beverage, in a pre-cursor to the beverage such as a beverage concentrate, or in ready-to-drink beverages. The antioxidant system. is particularly suitable for use in connection with ready-to-drink, coffee beverages and will be described primarily in this context. It is to be appreciated however that this is done for simplicity of description and the antioxidant system is not limited to this application.

[0018] The antioxidant system includes a glucose oxidase (EC 1.1.3.4). The glucose oxidase catalyses the oxidation of glucose to gluconic acid according to the following reaction scheme:

$$\text{Glucose} + O_2 + H_2O \xrightarrow[\text{oxidase}]{\text{glucose}} \text{gluconic acid} + H_2O_2$$

[0019] The antioxidant system also includes a catalase (EC 1.11.1.6). Then, the catalase degrades the peroxide according to the following reaction scheme:

$$H_2O_2 \xrightarrow{\text{Catalase}} 1/2\ O_2 + H_2O$$

[0020] The glucose oxidase and the catalase are preferably provided in the form of an enzyme mixture. A suitable enzyme mixture is the Novozym® 358 enzyme preparation commercialised by Novo Nordisk AS of Novo Allé, 2880 Bagsvaerd, Denmark. This enzyme preparation is prepared from *Aspergillus niger* and is generally recognised as safe.

[0021] The antioxidant system also includes a glucose oxidase substrate. This takes the form of glucose. The glucose oxidase substrate may be an inherent part of the beverage itself, may be added to the beverage, or both. In the case of coffee beverages, the glucose oxidase substrate is ordinarily added to the beverage mix because coffee contains little or no glucose. However, for other beverages which inherently contain glucose, further glucose need not be added.

[0022] The antioxidant system further includes an inorganic oxygen scavenger. Sulphites are particularly useful an inorganic oxygen scavengers. Suitable sulphites include sulphur dioxide, sodium sulphite, sodium metabisulphite, anhydrous sodium bisulphite, potassium metabisulphite, anhydrous potassium bisulphite, and mixtures of these agents. Sodium sulphite is particularly preferred. Apart from further removing oxygen, the inorganic oxygen scavenger removes hydrogen peroxide generated by the glucose oxidase.

[0023] The amount of the antioxidant system used will depend upon the substance to be treated and the level of oxygen present. Also, the amounts used of the various components in the antioxidant system will depend upon the substance to be treated and the level of oxygen present. Further, the amount of enzyme used will depend upon the activity of the enzyme. These amounts will be readily determined for each situation.

[0024] However, in general, the amount of glucose oxidase used is less than about 0.5% by weight of the total weight of the substance to be treated. For example, the amount of glucose oxidase used is preferably in the range of about 0.001% to about 0.1% by weight of the total weight of the substance to be treated. An amount in the range of about

0.005% to about 0.05% by weight is especially preferred for coffee beverages. The activity of the glucose oxidase is preferably about 1500 units/ml to about 2500 units/ml; for example about 2000 units/ml. A unit is the amount of enzyme which, at a temperature of 25°C and a pH of 5.1, catalyses the formation of 1 μmol of H_2O_2.

[0025] The amount of glucose oxidase substrate which is used is conveniently less than about 1% by weight of the total weight of the substance to be treated. For example, the amount of glucose oxidase substrate used is preferably in the range of about 0.005% to about 0.5% by weight of the total weight of the substance to be treated. An amount in the range of about 0.01% to about 0.3% by weight is especially preferred for coffee beverages; for example about 0.05% by weight. The glucose oxidase substrate may be present in the substance to be treated or may be added to the substance, or both. Usually, for coffee beverages, the glucose oxidase substrate is added. For beverages which must undergo heat treatment, the amount of glucose oxidase substrate used is preferably kept to the minimum necessary to obtain the required glucose oxidase activity. In this way, the formation of undesirable Maillard reactions may be avoided.

[0026] The amount of the inorganic oxygen scavenger which is used is conveniently less than about 0.1% by weight of the total weight of the substance to be treated. For example, the amount of inorganic oxygen scavenger used is preferably in the range of about 0.001% to about 0.05% by weight of the total weight of the substance to be treated. An amount in the range of about 0.002% to about 0.03% by weight is especially preferred for coffee beverages; for example about 0.005% by weight. Further, relevant regulatory requirements concerning the maximum residual levels of inorganic oxygen scavengers in foodstuffs should be respected.

[0027] If a catalase is used, the amount used is not critical. Usually the catalase will be provided in a mixture with the glucose oxidase and hence the catalase levels will be determined by the amount of glucose oxidase used.

[0028] The antioxidant system may be used at various points during the processing of a beverage. For example, for coffee and tea beverages, the antioxidant system may be added to the water which is to be used to extract soluble solids from the coffee or tea. In this way, the water which is used for extraction may be efficiently deoxygenated. However, because the glucose oxidase denatures at temperatures above about 60°C, the treatment should be carried out prior to heating the extraction water.

[0029] The antioxidant system may also be added to the extract obtained after extraction. At the time of addition of the enzyme of the antioxidant system, the temperature of the extract should be below about 60°C. After the extract has been deoxygenated, the extract may be thermally treated; for example during concentration or drying or both. The inorganic oxygen scavenger continues to operate at temperatures above 60°C. Of course, for best effect, all further processing of the extract should be carried out under oxygen reduced or oxygen free conditions. The various techniques described in the art may be used. In this way, a beverage, beverage concentrate or beverage powder which contains the antioxidant system and low levels of oxygen may be obtained.

[0030] The antioxidant system may also be added to the beverage prior to filling of the beverage into containers. At the time of addition of the enzyme of the antioxidant system, the temperature of the beverage should be below about 60°C. After the beverage has been deoxygenated, the beverage may be retorted in the usual manner. For best effect, the subsequent filling of the beverage into containers may be carried out under oxygen reduced or oxygen free conditions. The various techniques described in the art may be used. The beverage obtained preferably contains less than about 1 ppm of dissolved oxygen; more preferably less than about 0.5 ppm dissolved oxygen.

[0031] The antioxidant system may be used in combination with any type of beverage such as tea beverages, coffee beverages, chocolate beverages, malted beverages, and the like. However the system is particularly suited for use in coffee beverages since the system is able to compete with the potent antioxidant effects of coffee. Black coffee beverages, which are intended to have a clean, fresh flavour and aroma, are especially suitable. These beverages ordinarily contain about 0.5% to about 1.5% by weight of soluble coffee solids. They may also contain a sweetener.

[0032] Specific examples are now described to further illustrate the invention.

Example 1

[0033] Three beverages are prepared and are standardised to contain about 8 ppm of dissolved oxygen. The first beverage (beverage 1) is freshly brewed coffee which contains 1% by weight of soluble coffee solids. The second beverage (beverage 2) is prepared from a commercially available instant coffee and contains 1% by weight of soluble coffee solids. The third beverage (beverage 3) is freshly brewed coffee which contains 1% by weight of soluble solids, 0. 1% by weight of Novozym® 358 enzyme preparation, 0.1% by weight of glucose, and 0.008% by weight of sodium sulphite. The beverages are held in containers open to the ingress of air and the concentration of dissolved oxygen is determined at regular intervals.

[0034] The results are as follows:

Time (minutes)	Dissolved O_2 (ppm) Beverage 1	Dissolved O_2 (ppm) Beverage 2	Dissolved O_2 (ppm) Beverage 3
0	8	8	8
5	4.7	7.5	2
10	4.3	7.0	0.4
15	4.1	6.6	0.5
20	3.9	6.4	0.5
25	3.6	6.1	0.5
30	3.4	6.0	0.5
35	3.3	6.0	0.5
40	3.2	5.9	0.5
45	3.1	5.9	0.5
50	3.0	5.9	0.5
55	3.0	5.9	0.5
60	3.0	5.9	0.5

[0035] The results indicate the antioxidant system in beverage 3 removes dissolved oxygen much faster than freshly brewed and instant coffee. Therefore the antioxidant system is able to adequately compete with the coffee for oxygen; hence protecting the coffee from oxygen damage.

Example 2

[0036] Cans containing coffee solids are prepared. All cans contain about 1% by weight of coffee solids, about 5% by weight of sugar, about 0.065% by weight of sodium bicarbonate, and about 0.01% by weight of lysine. All cans are filled and sealed under the same conditions. During filling, the contents of each can are exposed to air.

[0037] Certain of the cans (the "Test cans") also contain an antioxidant system of 0.1% by weight of glucose, 0.01% by weight of Novozym® 358 enzyme preparation, and 0.005% by weight of sodium sulphite The other cans form a control (the "Control cans").

[0038] After 1 hour, 1 can from each group is opened and the dissolved oxygen is determined. The remaining cans of each group are then retorted and allowed to cool. After 12 days, a can of each group is opened and a sensory panel is used to analyse the aroma and flavour of the sample.

Group	Time (hours)	Dissolved O_2 (ppm)	Aroma & Flavour
Test	1	0.9	Fresh, clean flavours and aroma with roasty notes. Less acidity.
Control	1	6.8	Acid notes present. Prune-like, bland flavour.

[0039] The beverage of the test group has much less dissolved oxygen and much improved flavour and aroma.

[0040] Unopened cans of each group are stored for 10 weeks at room temperature and are then opened. The pH is determined. The beverage of the Control cans has a pH of about 5.5 while the beverage of the Test cans has a pH of about 5.7. A sensory panel is used to analyse the aroma and flavour of the beverage of the Test cans and it is found to have fresh, clean flavours and aroma.

Example 3

[0041] Roast and ground coffee is placed in an extraction system. The conditions are not oxygen free. The coffee is then extracted with one of three different types of deionised water at a temperature of about 25°C to 40°C. The first type, Type A, is untreated deionised water. The second type, Type 1, is deionised water which is treated with an antioxidant system of 0.05% by weight of glucose, 0.01% by weight of Novozym® 358 enzyme preparation, and 0.005% by weight of sodium sulphite. The third type, Type 2, is deionised water which is treated with an antioxidant system of

0.05% by weight of glucose, 0.1% by weight of Novozym® 358 enzyme preparation, and 0.005% by weight of sodium sulphite. The dissolved oxygen content of each type of deionised water and each extract is determined.

[0042] Each extract obtained is diluted with a sugar solution to provide a coffee beverage containing about 1% by weight of coffee solids. Each beverage is then filled into cans and the cans sealed. A can of each beverage is opened and the dissolved oxygen content of the beverage is determined. The remaining cans are retorted.

Water Type	O_2 Conc (ppm) in Extraction Water	O_2 Conc (ppm) in Extract	O_2 Conc (ppm) in Beverage
A	7.79	2.54	0.81
1	2.96	0.86	0.08
2	0.04	0.15	0.07

[0043] The results indicate that reducing the oxygen content of the extraction liquid greatly reduces the oxygen content in the resultant beverage, despite the beverage being produced under conditions which are not oxygen free.

[0044] Unopened cans of each group are stored for 10 weeks at room temperature and are then opened. A sensory panel is used to analyse the aroma and flavour of the beverages in the cans. The beverages produced using water Types 1 and 2 have a fresh, clean flavour and aroma. The beverages produced using water Type A have an unacceptable flavour and aroma.

Claims

1. A ready-to-drink beverage which includes an antioxidant system, the system comprising an enzyme composition containing a glucose oxidase, a glucose oxidase substrate and a catalase, and an inorganic oxygen scavenger.

2. A beverage according to claim 1 which contains about 0.001% to about 0.1% by weight of glucose oxidase.

3. A beverage according to claim 1 or claim 2 which contains about 0.005% to about 0.5% by weight of glucose oxidase substrate.

4. A beverage according to any of claims 1 to 3 in which the inorganic oxygen scavenger is a sulphite.

5. A beverage according to claim 4 which contains about 0.001% to about 0.05% by weight of sulphite.

6. A beverage according to claim 4 or claim 5 in which the sulphite is sodium sulphite.

7. A beverage according to any of claims 1 to 6 which is a black coffee beverage.

8. A beverage concentrate which includes an antioxidant system, the system comprising an enzyme composition containing a glucose oxidase, a glucose oxidase substrate, and a catalase, and an inorganic oxygen scavenger.

9. A process for reducing oxygen in a beverage, the process comprising:
adding an antioxidant system comprising glucose oxidase, a glucose oxidase substrate, a catalase and an inorganic oxygen scavenger to the beverage;

filling the beverage into containers; and
sealing the containers.

10. A process for reducing oxygen in a beverage containing extracted solids, the process comprising:

adding an antioxidant system comprising glucose oxidase, a glucose oxidase substrate, a catalase and an inorganic oxygen scavenger to an extraction liquid;
extracting solids from an extraction substrate using the extraction liquid to provide a beverage;
filling the beverage into containers; and
sealing the containers.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 99 20 0185

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
MUNICH	1 June 1999	Bendl, E

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Application Number
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